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POWERED BY **Dialog****METHOD AND APPARATUS FOR ANALYZING MINUTE AMOUNT OF OXYGEN IN METAL****Publication Number:** 10-073586 (JP 10073586 A) , March 17, 1998**Inventors:**

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**Application Number:** 08-248584 (JP 96248584) , August 30, 1996**International Class (IPC Edition 6):**

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**JAPIO Class:**

- 46.2 (INSTRUMENTATION--- Testing)

**Abstract:**

**PROBLEM TO BE SOLVED:** To perform analysis simply and quickly by removing the contaminated part of a sample by performing inactive-gas sputtering, substituting the atomospheric gas in a preliminary processing chamber to carrier gas for analysis, moving the sample into a heating chamber and analyzing oxygen.

**SOLUTION:** After Ar sputtering in a preliminary processing chamber 4 is completed, a sample 2 is moved into a reducing chamber 16 having a graphite crucible 14. Then, in order to obtain the same atmosphere as the carrier gas such as He in the same atmosphere as in the reducing chamber 16, Ar is changed to the He gas in the preliminary processing chamber 16. Then, the sample is charged as cathode and an outer frame 4A of the preliminary processing chamber 4 is charged as an anode through the conducting wire from a sputtering power supply 22. A high voltage is applied on the surface of the sample, and argon ions are made to callide. Thus, the contaminated part is broken away. A heating power supply 30 such as a high-frequency power supply is provided at the outside of the reducing chamber 16. The temperatrue of the preliminary processed sample is increased to the melting temperature of the sample 2 in the reducing atmosphere in the graphite crucible 14. Then, a trace of oxygen is determined as the CO gas by an infrared-ray absorbing device 32.

JAPIO

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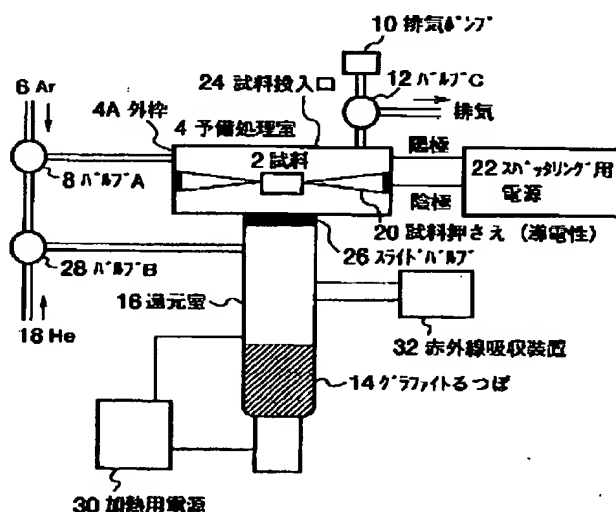
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(54) 【発明の名称】 金属中微量酸素の分析方法および装置

(57) 【要約】

【課題】 従来、金属中の微量酸素の分析方法および装置が種々開示されているものの、金属が純度が大きなるほど表面が大気中の有機物や炭酸ガス等に汚染されるので、汚染部を完全に除去した後でないと正確な酸素定量値を得ることができない。従来法は、この点について未だ満足すべきものはなかった。

【解決手段】 本発明は汚染部を除去するため、試料を不活性ガススパッタリングの予備処理を施すのを特徴とし、その後雰囲気ガスをアルゴン等の不活性ガスから分析キャリアガスのHe雰囲気に切り換え、下方に設けた分析装置中のグラフアイトるつぼを有する予備加熱室に移し、次に公知の酸素注出温度まで昇温し、酸素をCOガスとして公知の赤外線吸収装置にて定量する。



## 【特許請求の範囲】

【請求項1】 金属中微量酸素の分析方法において、試料を不活性ガス雰囲気の前記予備処理室に導入する段階と、前記試料に不活性ガススパッタリングを施し該試料の汚染部を除去する段階と、前記スパッタリング終了後前記予備処理室の雰囲気ガスを分析用キャリアガスに置換する段階と、前記予備処理室内の雰囲気ガスを分析用キャリアガスに切替えた後前記試料をグラフアイトするつぼを有する加熱室に移し酸素を分析する段階と、を有して成ることを特徴とする金属中微量酸素の分析方法。

【請求項2】 試料の表面汚染部を不活性ガスのスパッタリングにより除去する予備処理室と、前記スパッタリング用電源および不活性ガス源と、前記予備処理室の上部に開閉自在に設けられた試料投入口と、前記予備処理室へ導入されるスパッタリング用不活性ガス管および分析用キャリアガス管と、前記予備処理室の外側前方に設けられた前記不活性ガスおよび分析用キャリアガスの切換えバルブと、前記予備処理室を開閉自在に閉塞するシャッターと、前記予備処理室の直下に設けられ汚染部を除去された試料を移すグラフアイトするつぼを有する還元室と、前記還元室の加熱用電源と、前記還元室中の試料の酸素を定量する赤外線吸収装置を含む分析装置と、を有して成ることを特徴とする金属中微量酸素の分析装置。

## 【発明の詳細な説明】

## 【0001】

【産業上の利用分野】本発明は金属中微量酸素の分析方法および装置に係り、特に従来この種の分析方法および装置に比し簡便迅速にしかも高精度にて定量できる分析方法および装置に関する。

## 【0002】

【従来の技術】金属中の酸素は延性や加工性などの材料特性に影響を及ぼすために、正確な定量分析が必要である。特に近年、鋼の高純度化に伴い鉄鋼中の極微量の酸素が重要視されるようになり、その高精度でしかも迅速簡便な分析方法および分析装置が求められている。しかし、鋼の高純度化が進むと、各成分の含有量が低減されているため、その正確な分析には分析前に試料表面の汚染部を確実に除去することが極めて重要となっている。汚染源としては有機物や炭酸ガスの吸着、大気等の雰囲気による表面酸化物等が該当する。金属中の酸素含有量を正確に測定するにはこれらの汚染部を完全に除去した後分析する必要がある。

【0003】最近の鉄鋼中の微量酸素分析法としては、不活性ガス融解—赤外線吸収法が広く用いられており、試料表面の汚染部除去の前処理法としては、酸等を用いた電解研磨法や化学研磨法が報告されている。従来のこれらの方法の概要について説明する。

【0004】特開平3-150462号公報記載の発明の要旨は次のとおりである。すなわち、「鉄鋼中の微量

酸素を加熱抽出して測定する際に、前処理として試料の電解研磨を行なう方法において、酸素量測定前に1200℃以上1400℃以下の温度で予備加熱を行なうことを特徴とする鉄鋼の微量酸素分析方法。」である。すなわち、この発明は、試料表面に付着したガス、有機物等の汚染物を試料の酸素測定前に1200℃～1400℃の温度に加熱して除去し、鋼中に存在する微量酸素のみを分析せんとするものである。

【0005】特開昭60-18759号公報による発明の要旨は次の如くである。すなわち、「鋼中O<sub>2</sub>を分析するための供試料を反応管内に収容せしめると共に、該反応管内に還元性ガスを通入させ、該還元ガス雰囲気において500℃以上に加熱し前記供試料表面を還元処理することを特徴とする鋼中O<sub>2</sub>分析供試料の前処理法。」である。すなわち、この発明は、供試料を還元ガス雰囲気中で加熱することによって、試料表面の酸化膜を除去し、酸素分析の精度の向上を図らんとするものである。

【0006】しかし、上記従来法によって表面付着汚染を除去しても完全に酸化膜を除去することが困難であることが判明した。すなわち、上記発明の如き前処理を施しても、前処理の時点から分析計で測定するまでの時間帯に、試料洗浄等による試料表面汚染もしくは再酸化などにより試料表面が再度汚染されるためppmレベルもしくはそれ以下の定量分析に影響を与えることが明らかになった。特に清浄な鉄の表面は、雰囲気中の吸着や酸化が瞬時に起り易い。この問題を解決するために特開平6-148170号公報記載の発明は一つの方法を提案している。

【0007】この発明の要旨は、次の如くである。すなわち、「鉄鋼試料表面をグラインダー、ヤスリ等で研削後、該試料中の微量酸素を加熱抽出して測定する方法において、該研削処理後の試料を炭素ルツボに入れ900℃以上1,400℃以下の温度で予備加熱を行なうことを特徴とする鉄鋼中の微量酸素分析方法。」である。すなわち、この方法は、鉄鋼試料表面をグラインダー、ヤスリ等で研削後、試料を炭素ルツボに入れ、ヘリウム等の不活性ガス雰囲気中で900℃以上、1,400℃以下の温度で予備加熱する方法である。しかし、この方法はヘリウム等のキャリアーガス中でガス抽出する2,000℃前後の温度より低温で前処理するものであり、グラインダー等で研削された試料の表面付着酸素とルツボのCとの反応を前提としているものの、試料溶解前の上記低温では脱酸反応が起りにくく、しかもルツボに接触する部位のみに限定され、試料全体の脱酸反応を期待することができないという問題がある。

## 【0008】

【発明が解決しようとする課題】上記の如く、金属中特に鉄鋼中微量酸素の分析方法ならびに装置についての従来技術の欠点に鑑み本発明の目的は、簡便、迅速に分析

することができ、しかも高精度で信頼性の高い分析値を得ることができる効果的な金属中微量酸素の分析方法および装置を提供するにある。

#### 【0009】

【課題を解決するための手段】本発明による金属中微量酸素の分析方法および装置の要旨とするところは次の如くである。

(1) 金属中微量酸素の分析方法において、試料を不活性ガス雰囲気の前処理室に導入する段階と、前記試料に不活性ガススパッタリングを施し該試料の汚染部を除去する段階と、前記スパッタリング終了後前記予備処理室の雰囲気ガスを分析用キャリアガスに置換する段階と、前記予備処理室内の雰囲気ガスを分析用キャリアガスに切替えた後前記試料をグラフアイトのつぼを有する加熱室に移し酸素を分析する段階と、を有して成ることを特徴とする金属中微量酸素の分析方法。

(2) 試料の表面汚染部を不活性ガスのスパッタリングにより除去する予備処理室と、前記スパッタリング用電源および不活性ガス源と、前記予備処理室の上部に開閉自在に設けられた試料投入口と、前記予備処理室へ導入されるスパッタリング用不活性ガス管および分析用キャリアガス管と、前記予備処理室の外側前方に設けられた前記不活性ガスおよび分析用キャリアガスの切換えバルブと、前記予備処理室を開閉自在に閉塞するシャッターと、前記予備処理室の直下に設けられ汚染部を除去された試料を移すグラフアイトのつぼを有する還元室と、前記還元室の加熱用電源と、前記還元室中の試料の酸素を定量する赤外線吸収装置を含む分析装置と、を有して成ることを特徴とする金属中微量酸素の分析装置。

#### 【0010】

【発明の実施の形態】本発明の実施の形態を実施例に基づき図面を参照して説明する。図1は鉄鋼中の微量酸素の分析方法および装置を示す模式断面図である。先ず、本発明による分析装置について説明する。本発明の特徴は試料2の表面汚染部を不活性ガスのスパッタリングにより除去する方法をとり、不活性ガスとして主としてアルゴンを使用しアルゴンスパッタリングによって汚染部を除去する予備処理を行なうために予備処理室4を設けている。予備処理室4内ではArスパッタリングを完了するまではアルゴンガス源6からバルブ8を経由して予備処理室4に導入し、排気ポンプ10およびバルブ12によって排気させるように構成されている。

【0011】また、本発明法による予備処理室4におけるArスパッタリング完了後、試料2をグラフアイトのつぼ14を有する還元室16に移すために、還元室と同一雰囲気のHe等のキャリアガスと同一雰囲気にする必要上、予備処理室4内をArからHeへ切り換える。そのため予備処理室4の外側前方には切り換えバルブ8を経由してHeガス18が予備処理室4に導入できるようになっている。予備処理室4内の試料2はその全表面

の汚染部を除去する必要から試料2の支持は左右からの導電性試料押さえ20を介して点支持となっているのが特徴である。更にスパッタリングのため、スパッタリング用電源22からの導線を介して試料2は陰極に帯電し、予備処理室4の外枠4Aが陽極に帯電し、試料2の表面に高圧を印加しアルゴンイオンを衝突させることによって全表面の汚染部が破壊除去される構成となっている。

【0012】なお、予備処理室4の上部には、試料2の投入口24が設けられ、下部にはスライドバルブのシャッター26がいずれも開閉自在に設けられている。試料2のスパッタリングにより表面の酸素など不純物の除去終了後該試料の内臓されている酸素を分析のため、予備処理室4からグラフアイトのつぼ14を有する還元室16に移動させ空気に触れることなく、Ar雰囲気から分析用キャリアガスであるHeに切り換える。この切り換えはArガスの通路を閉とし、Heガスの通路を開とするバルブ8の切り換えによって行なわれる。予備処理室4のArからHeへの切り換え完了後、予備処理室4の底部のシャッター26を開とし、試料2を直下に設けられたグラフアイトのつぼ14を有する還元室16中に落下される。還元室16は分析室を形成するのでHe源18からバルブ28を経てキャリアガスHeが循環されている。

【0013】還元室16の外側には高周波電源等の還元室16の温度を調整できる加熱用電源30が設けられており、予備処理試料をグラフアイトのつぼ14の中の還元性雰囲気中で加熱される。還元室16の試料2は溶解する温度の2,000～2,500℃に昇温され、微量酸素はすべてCOガスとして公知の赤外線吸収装置32によって定量される。

#### 【0014】

【実施例】図2は試料2の表面をArでのスパッタリングした後の試料表面をオージェ電子分光法により分析した結果であり、横軸は電子エネルギーレベルを示し、縦軸はオージェ微分スペクトルを表す。図2より明らかとなり、スパッタリングした後の試料2の表層部にはほとんど酸素が観測されないことから酸化膜による汚染が完全に除去されていることを示す。

【0015】比較例として、図2に示したスパッタリングした後の同一試料を大気中に1分間放置した後、深さ方向に分析したオージェ電子分光法による分析結果を図3として示した。図3には高い値の酸素が検出されており、大気暴露により試料表面が再酸化されたことがわかる。

#### 【0016】

【発明の効果】本発明は金属中の特に鉄鋼中の微量酸素の分析に当り、従来法の欠点を一掃するため試料表面の汚染部の除去に、不活性ガススパッタリング現象を利用し、予備処理室において点支持の試料に対し、スパッタ

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【図2】本発明の実施例の一つとして、試料をアルゴンスパッタリングを施した後、オージェ電子分光法にて分析した。横軸は電子ボルトで表したエネルギーレベル、縦軸はオージェ微分スペクトルを示す線図で、試料表面には酸化膜による汚染が全くないことを示す。

【図3】図2に示す試料を大気中に1分間放置した後、深さ方向に分析した著大な酸素値を示す図2と同一のオージェ電子分光法によって表された線図である。

【符号の説明】

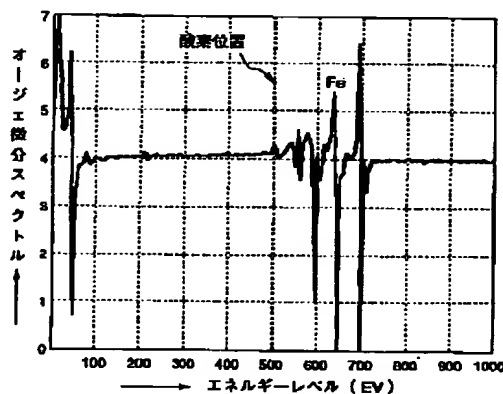
2	試料
4	予備処理室
6	アルゴンガス源
8、12	バルブ
10	排気ポンプ
14	グラフアイトるつぼ
16	還元室
18	He ガス
20	試料押さえ
22	スパツタリング用電源
24	試料投入口
26	シヤッター
28	バルブ
30	加熱用電源
32	赤外線吸収装置

10  
20

【図面の簡単な説明】

【図１】本発明による実施例として挙げた鉄鋼中微量酸素の分析方法および装置を模式系統的に示す線図である。

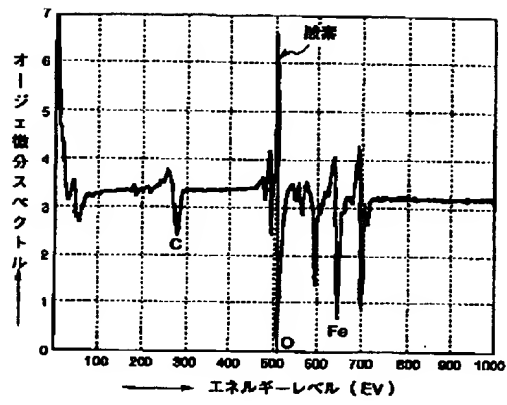
【図2】



(5)

特開平10-73586

【図3】



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CLAIMS

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[Claim(s)]

[Claim 1] The phase which introduces a sample into the preliminary processing room of the inert gas ambient atmosphere in the analytical method of the minute amount oxygen in a metal, The phase of performing inert gas sputtering to the aforementioned sample, and removing the pollution section of this sample, The phase of replacing the controlled atmosphere of the aforementioned account [ of sputtering end back to front ] preliminary processing room by the carrier gas for analysis, The analytical method of the minute amount oxygen in a metal characterized by having the phase of moving the account sample of back to front which changed the controlled atmosphere of the aforementioned preliminary processing interior of a room to the carrier gas for analysis to the heat chamber which has a graphite crucible, and analyzing oxygen, and changing.

[Claim 2] The preliminary processing room which removes the surface-contamination section of a sample by sputtering of inert gas, The aforementioned power for sputtering and the source of inert gas, and the sample input port prepared in the upper part of the aforementioned preliminary processing room free [ opening and closing ], The inert gas spool for sputtering and the carrier gas spool for analysis which are introduced at the aforementioned preliminary processing room, The aforementioned inert gas prepared ahead [ of the aforementioned preliminary processing room / outside ], and the switching bulb of the carrier gas for analysis, The reduction room which has the graphite crucible which moves the shutter blockaded free [ opening and closing of the aforementioned preliminary processing room ] and the sample which was prepared directly under the aforementioned preliminary processing room, and was removed in the pollution section, The analysis apparatus of the minute amount oxygen in a metal characterized by having the power for heating of the aforementioned reduction room, and an analysis apparatus containing the infrared-absorption equipment which carries out determination of the oxygen of the sample in the aforementioned reduction room, and changing.

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[Translation done.]



## \* NOTICES \*

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1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] this invention relates to the analytical method and equipment which relate to the analytical method and equipment of the minute amount oxygen in a metal, especially compare with this kind of a conventional analytical method and conventional equipment, and can moreover carry out determination with high degree of accuracy quickly simple.

[0002]

[Description of the Prior Art] The oxygen in a metal needs exact quantitative analysis, in order to affect material properties, such as ductility and workability. Especially, in connection with high-grade-izing of steel, importance comes to be attached to the oxygen of the ultralow volume in steel, and it is asked for the simple analytical method and simple analysis apparatus highly precise and quick moreover in recent years. However, if high-grade-ization of steel progresses, since the content of each component is reduced, it is very important for the exact analysis to remove certainly the pollution section on the front face of a sample before analysis. As a pollution source, the scaling object by the ambient atmosphere, such as adsorption of the organic substance and carbon dioxide gas and the atmospheric air, etc. corresponds. After removing these pollution sections completely to measure the oxygen content in a metal correctly, it is necessary to analyze.

[0003] As a minute amount oxygen analysis method in the latest steel, the inert gas synchysis-infrared absorption method is used widely, and an electrolytic polishing and chemical polishing using the acid etc. as a pretreatment method of the pollution section elimination on the front face of a sample are reported. The schema of such conventional technique is explained.

[0004] The summary of invention of publication-number 150462 [ three to ] official report printing is as follows. That is, it is "the minute amount oxygen analytical method of the steel characterized by performing a preheating before the amount measurement of oxygen in the technique of performing the electrolytic polishing of a sample, as pretreatment at 1200 degrees-C or more temperature of 1400 degrees C or less in case the heating extraction of the minute amount oxygen in steel is carried out and it is measured." That is, this invention heats and removes the contaminations adhering to the sample front face, such as gas and the organic substance, in temperature of 1200 degrees C - 1400 degrees C before oxygen measurement of a sample, and uses as an analysis plug only the minute amount oxygen which exists in steel.

[0005] the summary of invention by the Provisional-Publication-No. 18759 [ 60 to ] official report comes out as following That is, it is "the pretreatment method of the charge of O<sub>2</sub> analysis sample offering in steel characterized by carrying out \*\* ON of the reducing gas into this coil, heating at 500 degrees C or more in this reducing-gas ambient atmosphere, and carrying out reduction processing of the aforementioned charge front face of a sample offering while the charge of a sample offering for analyzing the inside O<sub>2</sub> of steel is made to hold in a coil." namely, the thing for which this invention heats the charge of a sample offering in the reducing-gas ambient atmosphere -- the oxide film on the front face of a sample -- removing -- the enhancement in the precision of oxygen analysis -- it is going to plan -- it is a thing

[0006] However, even if it removed surface adhesion contamination by the above-mentioned conventional method, it became clear that it is difficult to remove an oxide film completely. That is, even if it performed pretreatment like the above-mentioned from dawn, since a sample front face was again polluted by the time zone until it measures by the analyzer from the time of pretreatment by a sample surface contamination or a reoxidation by sample washing etc., it became clear to affect ppm level or the quantitative analysis not more than it. Especially as for a pure iron front face, adsorption and oxidization of the ambient atmosphere tend to happen at an instant. In order to solve this problem, invention of publication-number 148170 [ six to ] official report printing has proposed one technique.

[0007] the summary of this invention comes out as following That is, it is "a minute amount oxygen analytical method in the steel characterized by paying the sample after this grinding processing to a carbon crucible, and performing a preheating in the technique of carrying out the heating extraction of the minute amount oxygen after grinding and in this sample, and measuring a steel sample front face by the grinder, the file, etc. at 900 degrees-C or more temperature of 1,400 degrees C or less." That is, it is the technique of this technique putting a steel sample front face after grinding, putting it into a carbon crucible by the grinder, the file, etc., and carrying out the preheating of the sample in the inert gas ambient atmosphere, such as helium, at the temperature of 900 degrees C or more and 1,400 degrees C or less. However, this technique is what carries out pretreatment at low temperature from the temperature below and over 2,000 degrees C which carries out a gas extraction in carrier gas, such as helium. In the above-mentioned degree of low temperature before sample lysis of the thing on condition of the reaction of the surface adhesion oxygen of a sample and C of a crucible by which grinding was carried out by the

grinder etc., a deoxidation reaction seldom happens, it is limited only to the site which moreover contacts a crucible, and there is a problem that the deoxidation reaction of the whole sample is not expectable.

[0008]

[Problem(s) to be Solved by the Invention] Especially among a metal, in view of the analytical method of the minute amount oxygen in steel, and the fault of the conventional technique about equipment, the purpose of this invention can be analyzed simple and quickly and is like the above to offer the analytical method and equipment of the effective minute amount oxygen in a metal which can moreover acquire a reliable analysis value with high degree of accuracy.

[0009]

[Means for Solving the Problem] The place made into the analytical method of the minute amount oxygen in a metal by this invention and the summary of equipment is \*\* as following.

(1) The phase which introduces a sample into the preliminary processing room of the inert gas ambient atmosphere in the analytical method of the minute amount oxygen in a metal, The phase of performing inert gas sputtering to the aforementioned sample, and removing the pollution section of this sample, The phase of replacing the controlled atmosphere of the aforementioned account [ of sputtering end back to front ] preliminary processing room by the carrier gas for analysis, The analytical method of the minute amount oxygen in a metal characterized by having the phase of moving the account sample of back to front which changed the controlled atmosphere of the aforementioned preliminary processing interior of a room to the carrier gas for analysis to the heat chamber which has a graphite crucible, and analyzing oxygen, and changing.

(2) The preliminary processing room which removes the surface-contamination section of a sample by sputtering of inert gas, The aforementioned power for sputtering and the source of inert gas, and the sample input port prepared in the upper part of the aforementioned preliminary processing room free [ opening and closing ], The inert gas spool for sputtering and the carrier gas spool for analysis which are introduced at the aforementioned preliminary processing room, The aforementioned inert gas prepared ahead [ of the aforementioned preliminary processing room / outside ], and the switching bulb of the carrier gas for analysis, The reduction room which has the graphite crucible which moves the shutter blockaded free [ opening and closing of the aforementioned preliminary processing room ] and the sample which was prepared directly under the aforementioned preliminary processing room, and was removed in the pollution section, The analysis apparatus of the minute amount oxygen in a metal characterized by having the power for heating of the aforementioned reduction room, and an analysis apparatus containing the infrared-absorption equipment which carries out determination of the oxygen of the sample in the aforementioned reduction room, and changing.

[0010]

[Embodiments of the Invention] The gestalt of operation of this invention is explained with reference to a drawing based on an example. Drawing 1 is a type-section view showing the analytical method and equipment of minute amount oxygen in steel. First, the analysis apparatus by this invention is explained. The characteristic feature of this invention takes how sputtering of inert gas removes the surface-contamination section of a sample 2, and in order to perform preliminary processing which mainly removes the pollution section by argon sputtering as inert gas using an argon, it has formed the preliminary processing room 4. In the preliminary processing room 4, it introduces into the preliminary processing room 4 via a bulb 8 from the source 6 of argon gas until it completes Ar sputtering, and it is constituted so that it may be made to exhaust by the exhaust air pump 10 and the bulb 12.

[0011] Moreover, the inside of the preliminary processing room 4 in order to move a sample 2 to the reduction room 16 which has the graphite crucible 14, when [ required ] making it the same ambient atmosphere as carrier gas, such as helium of the same ambient atmosphere as a reduction room, is switched to helium from Ar after Ar sputtering completion in the preliminary processing room 4 by this invention method. Therefore, ahead [ of the preliminary processing room 4 / outside ] it switches, and helium gas 18 can introduce now into the preliminary processing room 4 via a bulb 8. It is the characteristic feature that support of a sample 2 is point support from the need that the sample 2 in the preliminary processing room 4 removes the pollution section of all the front faces, through the conductive sample presser foot 20 from right and left. Furthermore, for sputtering, through the lead wire from the power for sputtering 22, a sample 2 is charged in cathode, and when outer frame 4A of the preliminary processing room 4 is charged in an anode plate, impresses the hyperbaric pressure to the front face of a sample 2 and makes argon ion collide, it serves as the configuration that the destructive elimination of the pollution section of all front faces is carried out.

[0012] In addition, the input port 24 of a sample 2 is formed in the upper part of the preliminary processing room 4, and the shutter 26 of a slide valve is formed in the lower part by each free [ opening and closing ]. It switches to helium which is carrier gas for analysis from Ar ambient atmosphere, without making it move to the reduction room 16 which has the graphite crucible 14 from the preliminary processing room 4 for analysis of the oxygen to which the viscus of these samples after an elimination end of an impurity, such as surface oxygen, is carried out by sputtering of a sample 2, and touching air. This switch is performed by switch of the bulb 8 which makes the path of Ar gas close and makes the path of helium gas open. After switch completing from Ar to helium of the preliminary processing room 4, the shutter 26 of the pars basilaris ossis occipitalis of the preliminary processing room 4 is made open, and it falls all over the reduction room 16 which has the graphite crucible 14 in which the sample 2 was formed directly under. Since the reduction room 16 forms a chemical laboratory, carrier gas helium circulates through it through the bulb 28 from the source 18 of helium.

[0013] The power for heating 30 which can adjust the temperature of the reduction rooms 16, such as a RF generator, is formed in the outside of the reduction room 16, and a preliminary processing sample is heated by the reducing atmosphere in

the graphite crucible 14. The temperature up of the sample 2 of the reduction room 16 is carried out to 2,000-2,500 degrees C of the temperature to melt, and determination of all the minute amount oxygen is carried out by the well-known infrared-absorption equipment 32 as CO gas.

[0014]

[Example] Drawing 2 is the result of analyzing a sample front face after [ Ar ] carrying out sputtering for the front face of a sample 2 by the Auger electron spectroscopy, a quadrature axis shows an electronic energy level and an axis of ordinate expresses the Auger differential spectrum. A passage clearer than drawing 2 , since oxygen is hardly observed by the cortex section of the sample 2 after carrying out sputtering, it is shown that contamination by the oxide film is removed completely.

[0015] After leaving the same sample after [ which was shown in drawing 2 ] carrying out sputtering for 1 minute in the atmospheric air as an example of a comparison, the analysis result by the Auger electron spectroscopy analyzed in the depth orientation was shown as drawing 3 . The oxygen of a high value is detected in drawing 3 , and it turns out that the reoxidation of the sample front face was carried out by atmospheric-air exposure.

[0016]

[Effect of the Invention] this invention checked that remains oxygen did not exist in a front face at all by using an inert gas sputtering phenomenon for elimination of the pollution section on the front face of a sample, and performing sputtering to it to the sample of point support in a preliminary processing room, in order [ in a metal ] to sweep away the fault of a conventional method in analysis of the minute amount oxygen in steel especially. The effect like a degree was able to be mentioned for the equipment and technique of carrying out determination with the infrared-absorption equipment which moves the sample which does not almost have this pollution section into the graphite crucible of a reduction room, carries out a temperature up in elevated temperature of 2,000 degrees C or more, melts a sample, and makes all internal minute amount oxygen CO gas, for example, can be detected by the high sensitivity by \*\*\*\*\*s.

(b) The pollution section on the front face of a sample is not only completely removable, but although after sputtering is called instant, since it is not exposed into the atmospheric air, it is the big characteristic feature of this invention that there is no fear of a reoxidation, and the minute amount determination of oxygen in a metal can execute it with high degree of accuracy.

(b) Since the analytical method was comparatively easy, quantitative analysis became possible simple and quickly.

(c) By completion of this invention, it can contribute to a development of a high-grade material remarkably from now on.

[0017] In addition, it is clear that the technical thought of this invention can mainly do it about other metals other than steel although the example of this invention was explained about the analytical method and equipment of the minute amount oxygen in steel.

[0018]

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[Translation done.]